AFRL-RX-WP-TP-2009-4357

THIN FOIL TEST SYSTEM DEVELOPMENT (PREPRINT)

Ryan J. Morrissey, Garth Wilks, Michael Uchic, and Reji John

Metals Branch
Metals, Ceramics, and NDE Division

FEBRUARY 2009

AIR FORCE RESEARCH LABORATORY
MATERIALS AND MANUFACTURING DIRECTORATE
WRIGHT-PATTERSON AIR FORCE BASE, OH 45433-7750
AIR FORCE MATERIEL COMMAND
UNITED STATES AIR FORCE

NOTICE AND SIGNATURE PAGE

Using Government drawings, specifications, or other data included in this document for any purpose other than Government procurement does not in any way obligate the U.S. Government. The fact that the Government formulated or supplied the drawings, specifications, or other data does not license the holder or any other person or corporation; or convey any rights or permission to manufacture, use, or sell any patented invention that may relate to them.

This report was cleared for public release by the USAF 88th Air Base Wing (88 ABW) Public Affairs Office (PAO) on 02 Nov 2009 and is available to the general public, including foreign nationals. Copies may be obtained from the Defense Technical Information Center (DTIC) (http://www.dtic.mil).

AFRL-RX-WP-TP-2009-4357 HAS BEEN REVIEWED AND IS APPROVED FOR PUBLICATION IN ACCORDANCE WITH THE ASSIGNED DISTRIBUTION STATEMENT.

*//Signature//

RYAN MORRISSEY, Project Engineer
Metals Branch
Metals, Ceramics, & NDE Division

//Signature//

PAUL L. RET, Chief
Metals Branch
Metals, Ceramics, & NDE Division

//Signature//

ROBERT MARSHALL, Deputy Chief
Metals, Ceramics, & NDE Division
Materials and Manufacturing Directorate

This report is published in the interest of scientific and technical information exchange, and its publication does not constitute the Government's approval or disapproval of its ideas or findings.

*Disseminated copies will show "//Signature//" stamped or typed above the signature blocks.

REPORT DOCUMENTATION PAGE

Form Approved
OMB No. 0704-0188

1. REPORT DATE *(DD-MM-YY)*	2. REPORT TYPE	3. DATES COVERED *(From - To)*
February 2009	Technical Paper Preprint	01 January 2007 – 01 January 2009

4. TITLE AND SUBTITLE
THIN FOIL TEST SYSTEM DEVELOPMENT (PREPRINT)

5a. CONTRACT NUMBER
In-house

5b. GRANT NUMBER

5c. PROGRAM ELEMENT NUMBER
62102F

6. AUTHOR(S)
Ryan J. Morrissey, Michael Uchic, and Reji John (AFRL/RXLMN)
Garth Wilks (General Dynamics)

5d. PROJECT NUMBER
2510

5e. TASK NUMBER
01

5f. WORK UNIT NUMBER
25100102

7. PERFORMING ORGANIZATION NAME(S) AND ADDRESS(ES)
Metals Branch (AFRL/RXLM)
Metals, Ceramics, and NDE Division
Materials and Manufacturing Directorate
Wright-Patterson Air Force Base, OH 45433-7750
Air Force Materiel Command, United States Air Force

General Dynamics
Dayton, OH 45431

8. PERFORMING ORGANIZATION REPORT NUMBER
AFRL-RX-WP-TP-2009-4357

9. SPONSORING/MONITORING AGENCY NAME(S) AND ADDRESS(ES)
Air Force Research Laboratory
Materials and Manufacturing Directorate
Wright-Patterson Air Force Base, OH 45433-7750
Air Force Materiel Command
United States Air Force

10. SPONSORING/MONITORING AGENCY ACRONYM(S)
AFRL/RXLM

11. SPONSORING/MONITORING AGENCY REPORT NUMBER(S)
AFRL-RX-WP-TP-2009-4357

12. DISTRIBUTION/AVAILABILITY STATEMENT
Approved for public release; distribution unlimited.

13. SUPPLEMENTARY NOTES
PAO Case Number: 88ABW-2009-4609; Clearance Date: 02 Nov 2009. The U.S. Government is joint author of this work and has the right to use, modify, reproduce, release, perform, display, or disclose the work. Paper contains color.

14. ABSTRACT
The need to accurately determine the mechanical properties of thin foil materials has grown in recent years as the emphasis on hypersonic vehicles and access to space has also increased. In order to meet this need, two separate test systems have been developed in-house as part of a comprehensive program to advance state-of-the-art testing capabilities for thin foils. The first test system, developed using a conventional MTS hydraulic load frame outfitted with specialized capabilities, was designed for determining materials properties on a macro-scale, with specimen sizes on the order of 75-150 mm. The second system was designed to evaluate properties on a micro-scale, with corresponding specimen sizes on the order of 3 mm. This paper outlines the developmental process of each system, including unique challenges, preliminary data, and future considerations.

15. SUBJECT TERMS

16. SECURITY CLASSIFICATION OF:			17. LIMITATION OF ABSTRACT:	18. NUMBER OF PAGES	19a. NAME OF RESPONSIBLE PERSON (Monitor)
a. REPORT	b. ABSTRACT	c. THIS PAGE			Ryan J. Morrissey
Unclassified	Unclassified	Unclassified	SAR	20	19b. TELEPHONE NUMBER *(Include Area Code)* N/A

Standard Form 298 (Rev. 8-98)
Prescribed by ANSI Std. Z39-18

Thin Foil Test System Development

Ryan J. Morrissey[a], Garth Wilks[a,b], Michael Uchic[a], Reji John[a]

[a] US Air Force Research Laboratory, AFRL/RXLMN, Materials and Manufacturing Directorate, Wright-Patterson AFB, OH 45433, USA
[b] General Dynamics, Dayton, OH 45431, USA.

OVERVIEW

The need to accurately determine the mechanical properties of thin foil materials has grown in recent years as the emphasis on hypersonic vehicles and access to space has also increased. In order to meet this need, two separate test systems have been developed in-house as part of a comprehensive program to advance state-of-the-art testing capabilities for thin foils. The first test system, developed using a conventional MTS hydraulic load frame outfitted with specialized capabilities, was designed for determining materials properties on a macro-scale, with specimen sizes on the order of 75-150 mm. The second system was designed to evaluate properties on a micro-scale, with corresponding specimen sizes on the order of 3 mm. This paper outlines the developmental process of each system, including unique challenges, preliminary data, and future considerations.

MATERIAL

The initial material of interest for the Metallic Thermal Protection System (TPS) program was the nickel-base superalloy Haynes 230®. Haynes 230® is a solid solution strengthened alloy based on the Ni-Cr-Mo-W system. It is widely used in combustors, ducting, hot gas housings, stator casings and other static components used in land-based turbines. It is known for its high temperature strength, excellent oxidation resistance up to about 1150C and good long-term thermal stability [1].

The material was received in the form of three, twenty-five pound, 12.5” (31.75 cm) wide coils rolled to thicknesses 5, 10, and 20 mils (127, 254 and 508 μm, respectively). All of the material was rolled from the same starting ingot (heat code 83054782) and a comparison of the measured alloy composition to the baseline composition is given in Table 1. The rolling operations were performed by the Elgiloy Corporation of Elgin, IL.

Table 1: Comparison of the baseline (nominal) and actual alloy compositions.

	Ni	Cr	W	Mo	Fe	Co	Mn	Si	Al	C	La	B	Heat Code
baseline	57[a]	22	14	2	<3*	<5*	0.5	0.4	0.3	0.1	0.02	<0.015*	
All material	bal	22.546	14.178	1.299	1.245	0.224	0.544	0.376	0.381	0.108	0.015	0.002	830547842
	*Maximum	[a]As balance											

The Haynes 230® was received in the solution treated condition and the microstructure for all thicknesses consists of three primary components: (1) equiaxed, highly-twinned, gamma grains, (2) M_6C carbides throughout the matrix and (3) $M_{23}C_6$ carbides precipitated at the grain

boundaries. Figure 1 is an SEM image showing the equiaxed grain structure and the large M_6C carbides. The effects of rolling operations are evidenced by the stringered appearance of the M_6C carbides. The $M_{23}C_6$ carbides, while not visible in this image, decorate and highlight the visible grain boundaries in Figure 1.

The 20 mil thickness is attained through a single annealing/ rolling operation. The 10 mil thickness required two annealing/ rolling operations and the 5 mil required three. This difference in processing schemes for each thickness is demonstrated by a decrease in grain size as a function of annealing/ rolling operations. The effect of the annealing/ rolling operations is shown in Figure 2, a collection of electron backscattered diffraction (EBSD) image quality maps, where the grain size of the 5 mil material is finer than that of the 10 mil which is finer than the 20 mil material.

Figure 3 shows an EBSD pole figure map from the 20 mil (508μm) material. The annealing operations result in a fully recrystallized grain structure defined by the equiaxed grain shapes and general absence of any crystallographic texture. The results shown in figure 3 are typical for the 5 and 10 mil (127 and 254μm, respectively) material as well.

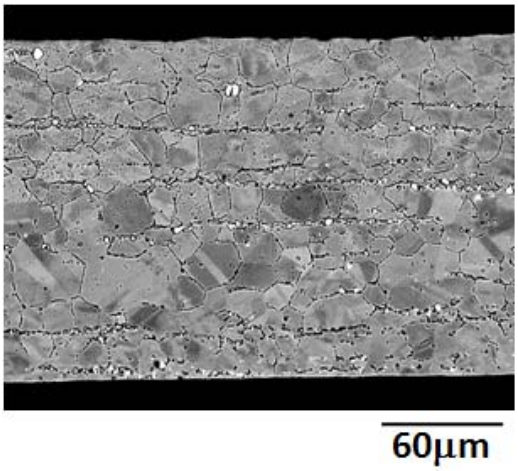


Figure 1: SEM images of typical solution treated Haynes230® microstructure. The equiaxed grain structure is evident as is the MC6 carbides (bright phase).

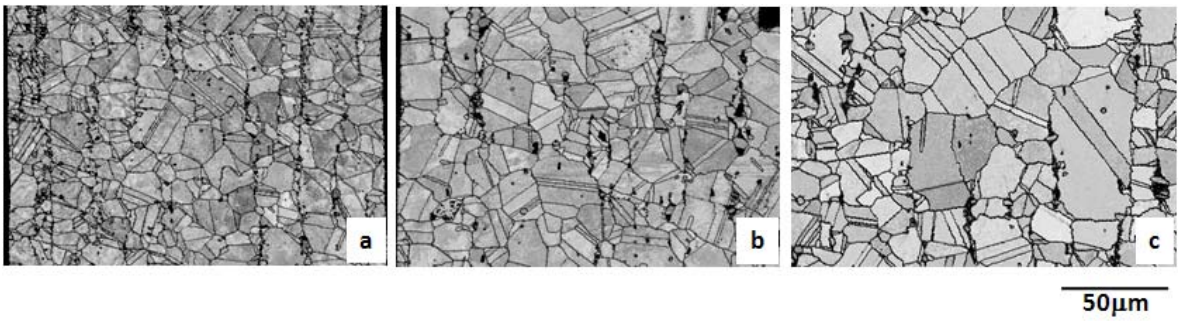


Figure 2: Comparison of the microstructures from the a) 5 mil - 127μm, b) 10 mil - 254μm and c) 20 mil -508μm material. Note the increase in grain size as material thickness increases.

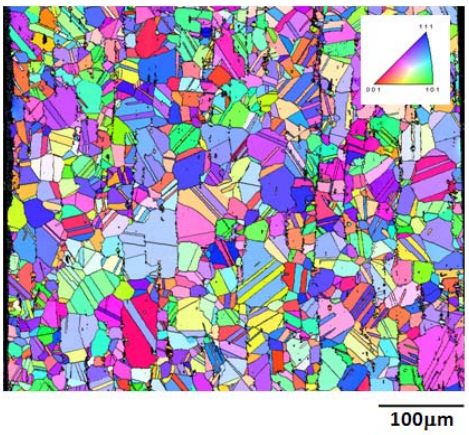


Figure 3: EBSD image of the 20 mil (508mm) material. Note the general lack of crystallographic texture in the material due to recrystallization. Twinning and the equiaxed nature of the material is also evident. Similar results, grain size aside, were observed for the 5 and 10 mil (127 and 254µm) material.

MACRO-SCALE TEST SYSTEM

The macro-test system is designed to determine mechanical properties of very thin foil (5-30 µm) using specimens of standard length (75-150 mm) and width (6-12 mm). The property values determined will be directly applicable to industry applications where thin foil materials are used over a large area, such as in thermal protection systems (TPS). A conventional MTS hydraulic load frame serves as the backbone of the system. A horizontal load frame, which eliminates the chimney effect when testing at elevated temperatures, was chosen for this system. However, very thin samples present several unique challenges which had to be overcome. The following sections describe the modifications that have been made to accommodate the unique requirements.

Specimen Preparation

The specimens for the macro-test system are flat dogbones with a total length of 152 mm and gage section minimum width of 8.50 mm, as shown in Figure 4. Due to the specimen thickness, it is not feasible to machine them directly. Instead, blanks are cut, stacked and secured between

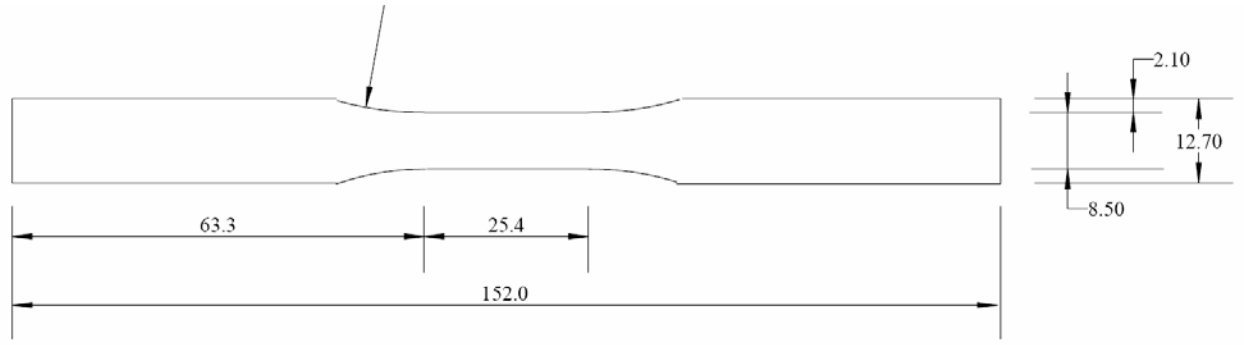


Figure 4: Specimen design

steel plates. Wire EDM is then used to machine the specimens to final dimensions. After machining, the stack is polished to remove burrs and machining marks from the specimen edges.

Specimen Alignment

Two fixtures were designed for the test system in order to minimize specimen misalignment and bending in the grips. The first, an alignment fixture designed with two micrometers attached to a small steel plate, was designed to ensure that the specimen was properly aligned with the grips and load train. The fixture, shown in Figure 5, is used each time a specimen is loaded in the test frame. The second fixture is used to eliminate out of plane motion by the grips. This alignment jig, shown in Figure 6, is designed to fit on top of the grip assembly and is also used each time a specimen is put installed. It is then removed prior to the start of the test.

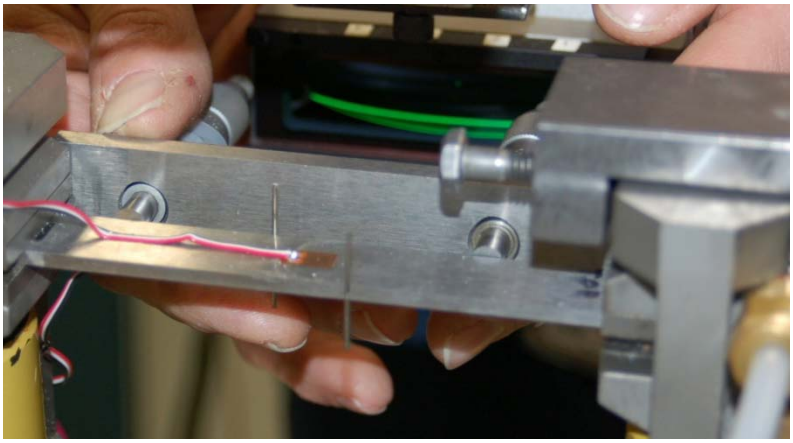


Figure 5: Specimen Alignment Fixture

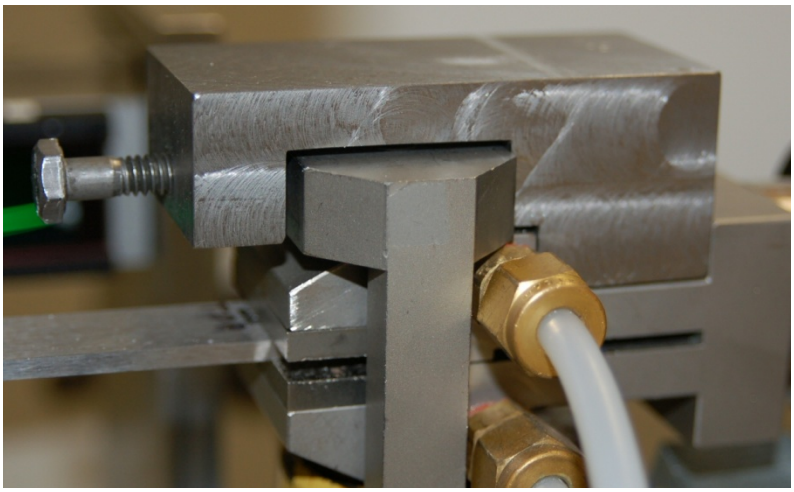


Figure 6: Grip Alignment Fixture

Displacement Measurement

Perhaps the most critical, and most difficult, aspect of testing thin foils involves the displacement measurement of the specimens. Due in part to the unique horizontal arrangement of the test system, the displacement in the gage section is measured using two vertical rods hanging from the specimen. These rods, or flags, rest on top of the specimen at a point, as well as along the side of the specimen (Figure 7). They allow the direct measurement of the displacement in the gage section between the two flags. Originally the system was designed using a laser micrometer to measure the flag movements, and therefore the displacement. The laser micrometer offers advantages such as good stability and exceptional accuracy (± 0.5 μm). However, it is limited by the data collection speed of 100 Hz. This slow speed causes problems during tensile tests run within ASTM standards by limiting the amount of data points collected. This becomes an even bigger issue when testing at elevated temperatures where creep effects will occur very quickly.

Due to the limitations of the laser micrometer system, a new digital optical micrometer was installed. This system uses a CCD array to provide continuous displacement data at over 2 kHz, as opposed to the linear scan of the laser system at 100 Hz. The digital system also provides two channels of data, which is very useful for separating high resolution data in the linear region from more periodic data over a wider displacement area during a complete tensile test. The validation of this system, using various combinations of displacement range, averaging, and scan rate, is currently nearing completion.

Elevated Temperature Testing

One significant goal of this project is to have the ability to test thin foil samples at elevated temperatures. Because of the unique test setup and displacement measuring system, a commercial-off-the-shelf (COTS) furnace was not available. Instead, a furnace was designed and built in-house. The furnace was specially designed with the following features:

1) Viewing ports on each side,

2) Four zone heating,

3) Small size to fit within restricted area of system,

4) Temperature capability exceeding 1200 C.

The viewing ports in item 1 are made using two optical quality quartz windows for each opening. This allows the optical micrometer to measure the displacement of the flags throughout each test. Four zone heating is accomplished using silicon nitride igniters, which have higher temperature capability and increased durability over silicon carbide igniters. Eight igniters are divided into four zones, with each zone controlled independently. This should allow for very stable, uniform temperatures across the specimen gage length. Initial testing of the furnace has verified temperature capability of 1200 C. Additional testing will be performed to verify and calibrate the system for accurate temperature control and uniform temperature across the specimen. The bottom half of the furnace assembly can be seen in Figure 7, as well as the micrometer looking through the quartz windows.

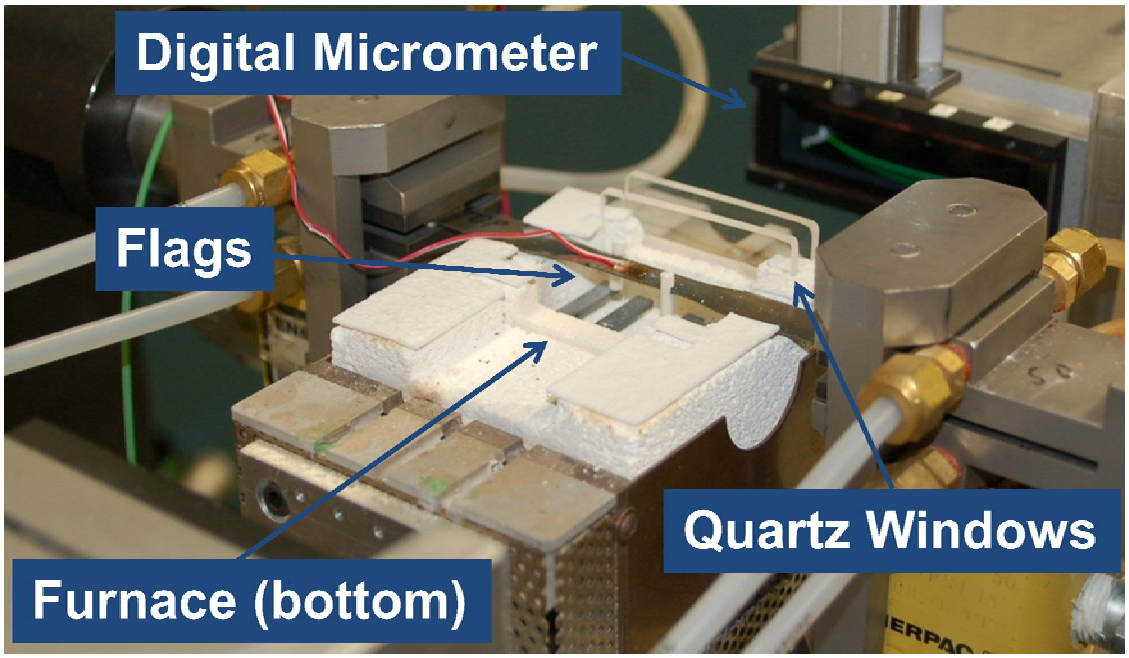
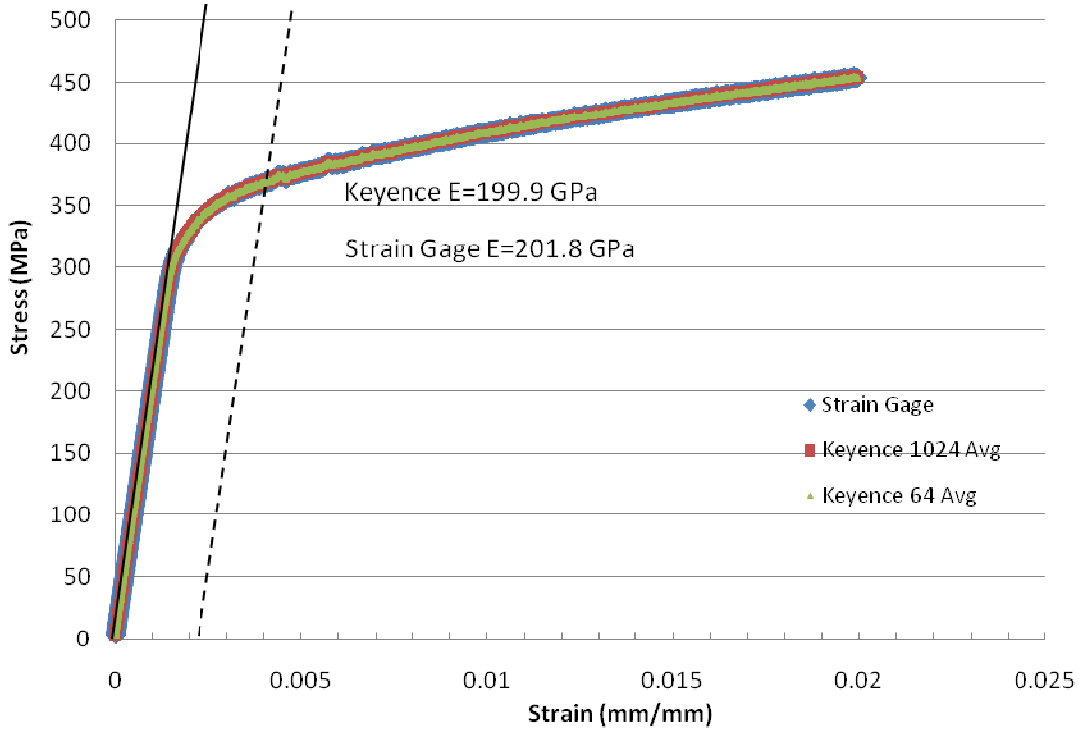


Figure 7: Test setup.

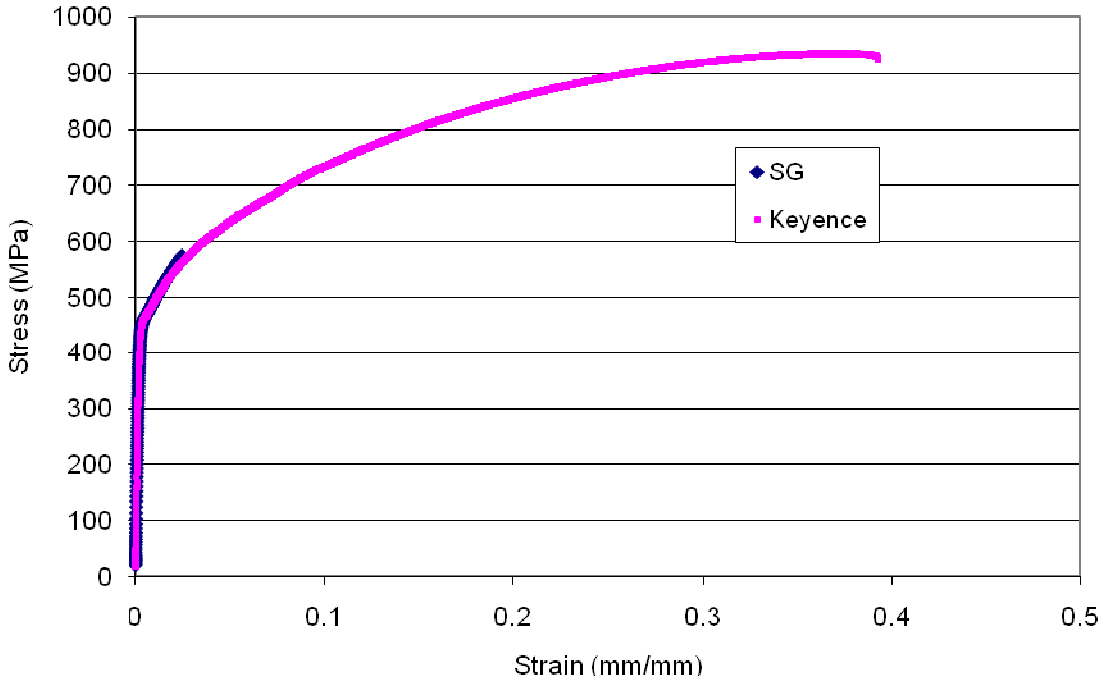
Initial Results

Extensive testing of the system components was performed to determine their operational capabilities. These tests were often performed using steel sheet material (0.02”-0.05” thick) to eliminate issues caused by the thin foils. Recently this phase of testing was completed for the room temperature setup, and initial tensile tests have begun using the Haynes 230 foil with thicknesses of 20 mil and 5 mil. The results of two of these tests are shown in Figure 8. Figure 8a shows the results of a tensile test on a 20 mil specimen out to the point where the strain gages failed (~ 2% strain). The modulus value determined using the strain gages and optical system are also shown on the plot and have very good agreement. Figure 8b shows the results of a

complete tensile test on a 5 mil specimen. In this case the recorded total strain exceeds 40%, much higher than is possible using strain gages.



a)



b)

Figure 8: Tensile tests on Haynes 230, a) 20 mil and b) 5 mil

MICRO-SCALE TEST SYSTEM

The micro-test system is designed to look at small quantities of material systems making it very useful for three specific types of applications. The first case is when mechanical property data (both room and high-temperature) is desired from small sections of components such as turbine-blade interstices or face-sheets for metallic thermal protection systems (TPS) in which the component dimensions limit the size of mechanical test specimens. Second, the mico-test apparatus is useful for assaying mechanical behavior in developmental material systems where only small quantities of material are available for experimentation (ex: single crystals of ordered intermetallics). Third, the system is useful as part of a suite of tools for measuring material properties over multiple length scales in order to understand size-dependent/length-scale affected material properties: those mainly dealing with microstructure homogeneity (second phase distribution, grain-size distribution, crystallographic texture, etc.).

The concept and operation of this system is generally very similar to a conventional (full-size) load frame with the exception that specimens (flat dog-bones) are on the order of 3 mm in length and ~300 microns in thickness. Also, due to the small size of the gauge area, it is difficult to instrument such specimens with strain gauges and thermocouples, so displacement will be measured via computer-controlled interferometric strain gauges on either face of the dog-bone while temperature is to be measured via a small-spot size pyrometer. Specimen heating is done resistively through Ni-based grips that are electrically insulated from the rest of the frame. Strain rates that can be applied by this system are limited to quasi-static (~10^{-3} ε/s) due to the micro-threaded screw drive used to apply load, which itself is limited to ~25 lbs force.

Work to date on this system has focused on removing impediments to room-temperature tests, as well as the execution of several such tests while simultaneously retrofitting the system for high temperature capability. Preparing the system for room temperature testing involved determining the origin of errors in displacement measurements via the interferometric strain-gauges as well as developing a fixture for controlling specimen alignment. Preparation of the micro-test system for high-temperature testing involved designing and machining superalloy (PW1484) grips, ceramic insulating components, as well as the purchase of resistive heating components and a pyrometer for measuring specimen temperature. The details of each of these issues are discussed in this report.

Room Temperature Mechanical Behavior

Alignment

Because of the fixed mount in the original system, positioning specimens was difficult. This affected both the proper measurement of displacement with interferometric strain gauges (via the alignment between specimen indents and the detectors), as well as the mechanical alignment of and bending stresses in the specimen. Unfortunately, as tensile specimens are decreased in size, small misalignments can introduce large bending moments and misrepresent the uniaxial yield behavior by a substantial amount. The figure below illustrates the effect of several orders of misalignment (1, 10, and 100 microns) on the measured flow stress in a uniaxial tension test as a function of specimens (sheet) thickness.

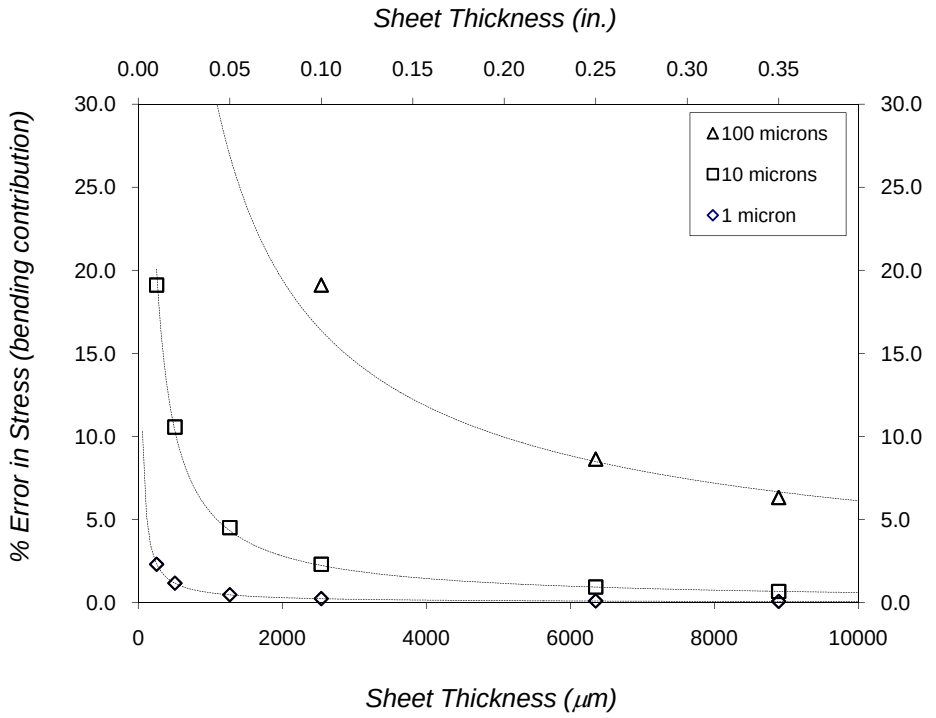


Figure 9: Effect of Specimen Alignment/Eccentricity on Stress Measurement

It was observed that the initial fixed-grip system, a virtual clone of the original system used at Johns Hopkins University (JHU), could control specimen loading eccentricity/alignment to, on the order of 100 microns. Since this system was originally conceived to measure the slip character of single crystals, and that is ultimately one of the intended uses for this system as well, it was felt that an error on the order of 20-30% in yield strength measurement (Fig. 9) would overwhelm variations in slip character for different orientations of single crystal superalloys, and was unacceptable.

In response to this issue, it was necessary to build a CAD model of the existing system to be able to design, purchase, and machine fixtures that could control specimen alignment. This model is pictured below, Figure 10. The main modification implemented to control alignment was the replacement of the fixed grips with a 6-axis positioner at the left end of the rig. This fixture, designed by Elliot Scientific, was purchased from a retailer of their products, Lightspeed Technologies and attached to the system in conjunction with a redesign of the grips that ensured compatibility.

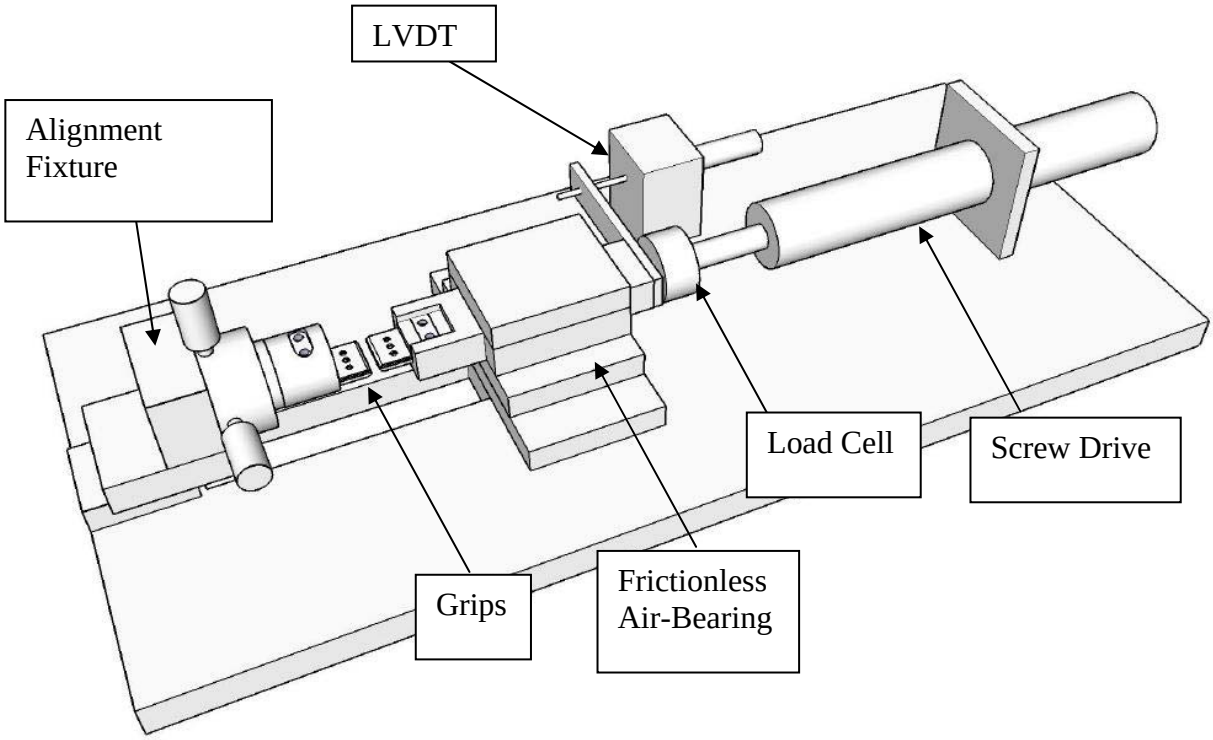


Figure 10: CAD model of principal micro-test system components with new alignment fixture.

Displacement Measurement

Displacement is measured in the system via an interferometric strain gauge technique. In this technique, two hardness indents are placed on both sides of the specimen in the gauge section to diffract incident laser light into fringe patterns that are detected with a total of 4 Si-detectors (two on each side). This system was mainly implemented using Marc Zupan's dissertation as a template, including the software controlling the measurement of displacement via a Fourier technique that interpreted the evolution of these diffraction fringes. Though this technique was implemented verbatim from the dissertation, a typo resulted in the incorrect calculation being used to measure displacement/strain. Explicitly, the displacement measured by the pair of detectors on one side of the specimen has two components, the strain in the specimen *and* rigid body motion; the dissertation indicated that specimen strain could be determined by averaging the readings from both detectors on a side, when in fact, it was necessary to take the difference between the two. The software was corrected so that strain could be correctly measured.

Room Temperature Test Results

Prior to final modifications to the alignment fixture, several room temperature tests on specimens machined from Haynes 230 were run to assay the efficacy of the system. Stress-strain results for two tests are depicted in Fig. 11. The three traces for each specimen represent the front-face and back face measured strain as well as their average.

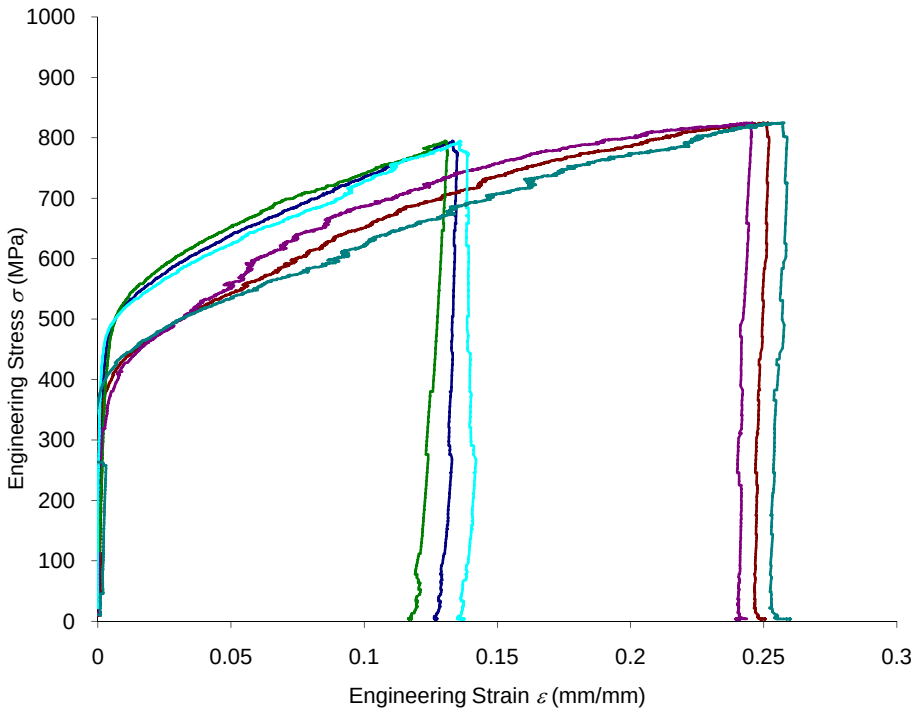


Figure 11: Engineering Stress-Strain Results for Haynes 230 micro-test specimens.

Modulus, yield strength and strain hardening characteristics are commensurate with literature data for this material. Total displacement was arbitrary. Variation between the two specimens could be the result of variations in specimen alignment as well as microstructure.

High Temperature Testing

Readying the system for high-temperature mechanical behavior measurement has been a concurrent focus of this work. To this end most if not all necessary equipment has been acquired.

A networkable precision power supply from Agilent has been obtained to resistively heat the specimen. Control of this power supply is currently being integrated into the existing Labview control software used to control the motion of the screw-drive and measure displacement.

A pyrometer (MIGA-12, MBE23) has been purchased from Mikron to measure temperature; this pyrometer has a spot size of 250 microns, less than thickness of the specimen, with interchangeable optics that will permit a spot size of down to 100 microns. This mitigates the approximations used in the JHU set-up to measure temperature which was caused by only having a pyrometer with a spot-size greater than their specimen dimensions—another significant improvement in the confidence of results that will be produced by this system over those of the JHU system. The 250 micron spot size optics were selected initially because the working distance for these lenses keeps the pyrometer 6” away from the hot specimen where the smaller spot size would require moving the pyrometer within 3” of the hot specimen, an arrangement that would most likely require active cooling of the pyrometer. This pyrometer will be integrated into a feedback loop to control temperature via the power supply. Positioning fixtures for the pyrometer have also been purchased.

A new grip assembly has been designed for the system so that specimens can be resistively heated (Fig. 12). We explored the possibility of using a low resistivity material for the grips since it is believed that the relatively low conductivity of a Ni-based alloy will heat the grips more than if the grips were made out a high-conductivity Cu-based system (Narloy, Glidcop, etc.), however, it was felt that these materials may limit the high temperature capability of the system because of their relatively poor creep performance. Consequently, PWA1484 has been used to machine high-temperature grips. These grips are insulated from the rest of the frame by components machined from a glass-matrix ceramic that goes by the trade name of MACOR.

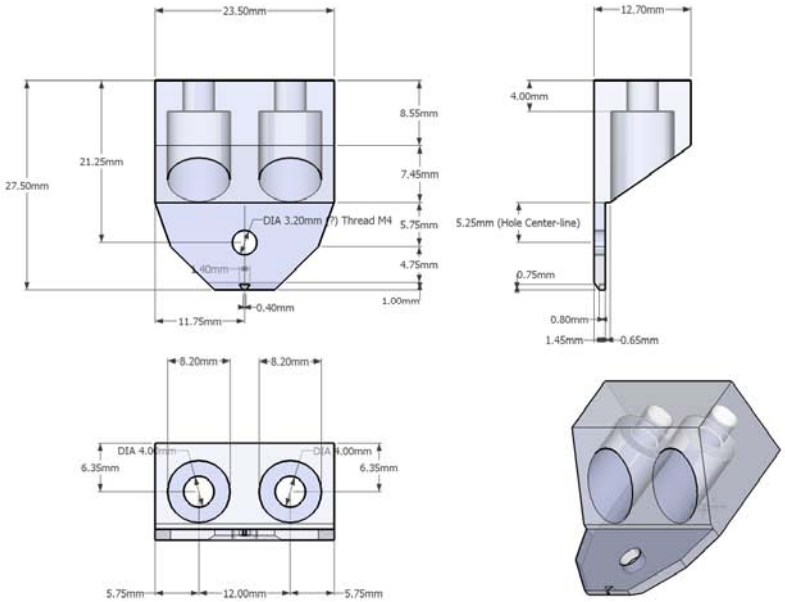


Figure 12: High Temperature PW1484 Grips

Integration of these components is proceeding, and a high-temperature proof-of-concept test is imminent. After which, measurement techniques will be verified and validated, and then the remaining lot of Haynes 230 will subsequently be tested and compared to the results of larger scale tests on the same material.

Verification & Validation

Regarding displacement measurement, after surveying the several vendors that provide optical displacement/strain measurement systems, the capabilities of the systems available were dissapointing--as of yet the state-of-the-art systems do not appear capable of measuring strain at high temperature to a sufficient resolution for this apparatus. The prototype system that the life-prediction group has ordered is the closest realization of that desire, and it must resort to some methods which may be impractical for this system. However, a video displacement system will be very useful for this system in order to initially align the specimen and measure displacement at low temperatures. To that end, a short-focal length lens will be purchased and used in conjunction with the ceramics group's video-displacement system to measuring the uniformity of displacement and the presence of undesired bending stresses.

Temperature measurement and control will be verified through several specimens that have been ordered with micro-sized thermocouples welded to their surface. Relays have been purchased that will be integrated into control system to rapidly shut-off and turn-back-on the resistive current heating the specimen so that a temperature measurement can be made via thermocouple for comparison with pyrometer results.

Future Considerations

The screw-drive in the current system strongly limits the strain-rate and load that can be applied to a specimen and has also proven to be only intermittently reliable and difficult to align and reassemble after design iterations. The company that produces the drive is no longer in business. In the future a piezo-electric stack should be appropriated and integrated into the system to provide variable-rate displacement and higher load capacities for this system. This would strongly augment the robustness of the system, and allow for broader exploration of the deformation mechanism-space for high-temperature material systems.

References

[1] Haynes on-line literature, No. H-3000, Haynes 230 Alloy Brochure, www.haynesintl.com.

[2] Zupan, Mark, "Microsample characterization of the tensile and compressive mechanical properties of single crystalline gamma-TiAl", Ph.D. dissertation, Johns Hopkins University, 2000.